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METHOD 531.1

I. SCOPE AND APPLICATION:

This is a high performance liquid chromatographic (HPLC) method applicable to the determination of certain N-methylcarbamoyloximes and N-methylcarbamates in groundwater and finished drinking water. The following compounds can be determined using this method:

Analyte	Chemical Abstract Services Registry Numbers (CASRN)
Aldicarb	116-06-3
Aldicarb sulfone	1646-88-4
Aldicarb sulfoxide	1646-87-3
Baygon	114-26-1
Carbaryl	63-25-2
Carbofuran	1563-66-2
3-Hydroxycarbofuran	16655-82-6
Methiocarb	2032-65-7
Methomyl	16752-77-5
Oxamyl	23135-22-0

II. REAGENTS:

- Sodium Thiosulfate ($Na_2S_2O_3$) solution
- Monochloroacetic acid buffer solution

III. MATERIALS:

- 60 ml amber borosilicate sample bottle fitted with screw caps lined with TFE-fluorocarbon.
- Latex gloves
- Paper towels
- Plastic container for disposal of used pipette tips
- Disposable glass pipette and rubber bulb.
- Kim wipes
- Pliers
- Protective Eyewear

IV. PROCEDURE:

1. Remove any attachments such as hoses, screens or aeration devices on the faucet. Inspect the faucet for anything that may fall into the sample container.

2. Open the tap and allow the system to flush for about 10 minutes. This should be sufficiently long enough to allow the water temperature to stabilize and get a representative sample.

3. Adjust the water flow to about 500 ml/minute or slow enough that no air bubbles purge the sample when collecting from the flowing stream.

4. Remove the cap from the 60 ml container. Do not rinse the container as it has already been acid rinsed and may already contain monochloroacetic acid buffer as a preservative.

5. To fill, tip the bottle to about a 45° angle into the stream of water. Ensure the stream is sufficiently slow so as to be able to anticipate when the bottle is nearly full and thus avoid over flowing. Fill the bottle to within approximately ½ inch of the mouth.

6. Remove the bottle from the flow and recap. Invert the sample bottle three times.

7. Place a chlorine detector strip on a dry opened paper towel. Remove the screw-on cap and obtain an aliquot of the sample using a glass pipette. Moisten the chlorine detector strip with the aliquot from the glass pipette and immediately flick the chlorine detector strip once using a sharp wrist motion to shake off the excess water. Compare the strip with the reference chlorine range. A determination must be made within 30 seconds.

IV. PROCEDURE (continued):

8. If no chlorine is detected, recap the bottle firmly, dry the sample bottle, attach the sample/laboratory label to the bottle and secure the chain of custody seal around the cap. Record the results in the field notebook and place the sample bottle in the ice chest to cool to 4°C.

9. If chlorine is present, add 5 drops of sodium thiosulfate solution, recap the bottle firmly and invert three times. Place a chlorine detector strip on a dry opened paper towel. Remove the screw-on cap and obtain an aliquot of the sample using a glass pipette. Thoroughly moisten the chlorine detector strip with the aliquot from the glass pipette and immediately flick the chlorine detector strip once to shake off the excess water. Compare the strip with the reference chlorine range.

10. If no chlorine is detected, recap the bottle firmly, dry the sample bottle, attach the sample/laboratory label to the bottle and secure the chain of custody seal around the cap. Record the results in the field notebook and place the sample bottle in the ice chest to cool to 4°C.

11. Continue the process of adding sodium thiosulfate to the sample, recapping, mixing, and testing until no chlorine is detected. Remember to note the number of drops of sodium thiosulfate added to the water sample in the field notebook.

V. SAMPLE TRANSPORT:

After obtaining the water samples, attach the completed chain of custody seal around the plastic cap of each 1-liter bottle. The 1-liter bottle must be amber colored to reflect sunlight since some of the pesticides analyzed for in this method are light sensitive and degrade when exposed to ultraviolet radiation. Place the sample bottle into the ice chest for transport. The samples must be chilled and preserved at a temperature of 4°C and maintained at that temperature until analysis. Always use chopped, grated, or dry ice when chilling the voa samples for transportation. Never use “blue ice” as the samples will not adequately chill. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure they will be at 4°C upon arrival at the laboratory.

VI. SAMPLE PRESERVATION:

The samples must be iced or refrigerated at 4°C and protected from light from the time of collection until extraction. Limited holding studies have indicated that the analytes thus stored are stable for up to 14 days or even longer if preserved properly.

VII. DEFINITIONS:

A. *Sodium Thiosulfate ($Na_2S_2O_3$):* A preservative use to dechlorinate water samples. Reduces free chlorine into acid.

VIII. SAFETY:

The use of protective eyewear and laboratory quality latex gloves is highly recommended when collecting and preserving samples.

IX. SUMMARY OF METHOD:

METHOD 531.1: The water sample is filtered and a 400 µl aliquot is injected into a reverse phase high performance liquid chromatography (HPLC) column. Separation of the analytes is achieved using gradient elution chromatography. After elution from the HPLC column, the analytes are hydrolyzed with a 0.05 N sodium hydroxide (NaOH) at 95°C. The methylamine formed during hydrolysis is then reacted with o-phthalaldehyde (OPA) and 2-mercaptoethanol to form a highly fluorescent derivative which is detected by a fluoresence detector.